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Prepare Mineral Samples for Analysis

Mineral dressing

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Terminology

- Heterogeneous
  - A substance that doesn’t have the same consistency throughout
  - Can cause variations and bias in analytical results
- Homogeneous
  - A substance that has the same consistency throughout
  - Important in mineral analysis to ensure minimal variation or bias in analytical results
- Integrity
  - Vitally important to ensure all samples integrity is maintained from collection to disposal during the mineral preparation process
- Segregation
  - The ability to separate minerals within a sample due to their physical or chemical properties e.g. sample particle size, magnetic properties

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Terminology

- Ore
  - An ore is natural rock or sediment that contains one or more valuable minerals, typically containing metals, that can be mined, treated and sold at a profit. Ore is extracted from the earth through mining and treated or refined, often via smelting, to extract the valuable metals or minerals. Wikipedia
- Gangue
  - In mining, gangue is the commercially worthless material that surrounds, or is closely mixed with, a wanted mineral in an ore deposit. Wikipedia
- Refractory
  - A refractory material or refractory is a material that is resistant to decomposition by heat, pressure, or chemical attack, and retains strength and form at high temperatures. Refractories are polycrystalline, polyphase, inorganic, non-metallic, porous, and heterogeneous. Wikipedia
    - e.g. magnetite (Fe_3O_4), ilmenite ($FeTiO_3$), chromite ($FeCr_20_4$), zircon ($Zr0_2$)...
- Analyte
  - An analyte, component, or chemical species is a substance or chemical constituent that is of interest in an analytical procedure. The purest substances are referred to as analytes. Example: 24 karat gold, NaCl, water, etc. Wikipedia

Sample types

- Sulfides
  - One or more chemical elements combined with sulphur e.g. pyrite FeS_2 (fools gold)

https://commons.wikimedia.org/wiki/File:Pyrite_3.jpg

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Sample types

- Oxides
  - One or more chemical elements combined with oxygen e.g. hematite, Fe_2O_3

https://upload.wikimedia.org/wikipedia/commons/2/24/Hematite-rich_iron_ore_%28Precambrian%3B_Pioneer_Mine%2C_Ely%2C_Minnesota%2C_USA%29_3.jpg

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Sample types

- Silicates
  - One or more chemical elements combined with silicon and oxygen e.g. mica, (muscovite), $KAl_2(Si_3Al)O_{10}(OH,F)_2$

https://commons.wikimedia.org/wiki/File:Mica_-_Silicate_Mineral.jpg

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Typical materials sampled

- Solids
  - Rocks
    - The solid mineral forming part of the surface of the earth and other similar planets, exposed on the surface or underlying the soil (Oxford dictionary)
  - Minerals
    - In geology and mineralogy, a mineral or mineral species is, broadly speaking, a solid chemical compound with a fairly well-defined chemical composition and a specific crystal structure that occurs naturally in pure form. Wikipedia
  - Soils
    - Soil, also commonly referred to as earth or dirt, is a mixture of organic matter, minerals, gases, liquids, and organisms that together support life. Wikipedia
  - Sands
    - Sand is a granular material composed of finely divided rock and mineral particles. It has various compositions but is defined by its grain size. Sand grains are smaller than gravel and coarser than silt. Wikipedia
  - Stream sediments
    - Streambed sediments are fine mineral deposits and silt located on the streambed. Google definition

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Typical materials sampled

- Pulverised core samples
  - Core samples typically jaw crushed and pulverised to a fine powder prior to analysis
- Powder concentrates
  - Matte samples
  - Mixed sulphide (nickel, copper, cobalt sulphides)
- Dump samples
  - Sampling from dump trucks or waste heaps
- Grab samples
  - A grab sample is a single sample from one location using a scoop or another container. Any layering of materials is destroyed in the sampling process, offering a mixed sample for testing. This single sample offers a discrete snapshot of a single spot in the sampling area.
- Composite samples
  - Composite samples consist of multiple grab samples taken over an area or time period.

Typical materials sampled

- Drill samples: (Wikipedia definitions)
  - Rotary air blast (RAB)
    - Rotary Air Blast drilling, is **an** open-hole technique in which compressed air is injected down the drill pipe in order to recover the cuttings up the outside of the drill stem to the surface
  - Reverse circulation (RC)
    - Reverse Circulation (RC) drilling is one of the three main types of mineral exploration techniques offered by Ranger. Used to compile sample rock cuttings, the drilling process relies on hollow inner tubes to transport samples back to the surface in a continuous flow, using compressed air.
  - Diamond drilling
    - Diamond Drilling is a form of core drilling which uses a rotary drill with a diamond drill bit attached in order to create precisely measured holes. As diamond is the hardest naturally occurring material in the world, it is ideal for creating openings in a range of materials including concrete, metal and glass.
  - Aircore
    - Air Core drilling utilises high-pressure air and dual walled rods to penetrate the ground and return the sample to the surface through the inner tube and then through a sampling system. The ground is cut through with the use of a steel blade type bit. This sample is then collected and analysed.
- NOTE: What is the difference between RC and diamond drilling?
  - Diamond drilling is more efficient for precise sampling and analysis, whereas RC drilling is adept at extracting bulk samples. When it comes to speed, RC drilling is the faster method.

Sample packing

- Samples arrive at a laboratory usually via courier truck or van from a mine or exploration site
- The samples are packed in cloth or calico bags stored inside large plastic bags
- Each plastic bag may hold up to 10 samples each, depending on the amount of sample in each sample bag
- The plastic bags are packed from site into a large boxed crate for transfer by forklift to a truck for transport to the laboratory for preparation and analysis
- Once at lab arrival, samples are offloaded and sorted and cross checked against the supplied chain of custody information to ensure all samples are present and accounted for.
- Once sorted, the samples are numerically placed into drying racks before being prepared for analysis

https://tigertek.com.au/products/composite-drawstring-poly-cotton-blended-calico-bag-plain-copy/

Sample Preparation methods

- Mineral dressing
  - sorting, boxing and drying
    - Samples first need to be sorted, typically numerically or in sequence as per client paperwork
    - Samples are dried in a drying oven if they are soil or ore samples. Damp or wet samples will stick to preparation equipment leading to contamination issues between samples
    - After preparation and prior to lab submission for analysis, samples are placed into sample bags and then boxed in batches for analysis
  - Sieving
    - Separation of the sample due to its particle size using sieves of decreasing hole size
  - Primary crushing (e.g. 10mm, 2mm)
    - Large size samples preliminary crushed using typically jaw crushers to achieve a particle size of between 10mm down to 2mm
  - Fine crushing (e.g.100 micron, 75 micron)
    - Secondary crushing to reduce particle size to between 100 and 75 µM
    - In most cases this may involve another crushing device (e.g.ball or roller mill) or a ring mill pulveriser
    - Aim to achieve a consistency of talcum powder

Function of key components and operating procedures for sample preparation equipment

- Jaw crushers
  - Used as particle size reduction device
  - Typical size crushing to <2mm
  - Used to crush:
    - minerals/ores/soil/slags/drill core/
    - cements/clinker/concrete/
    - glass/ceramics/sludges/coal/coke/industrial waste
  - Video
    - https://images.app.goo.gl/VUwQ9vFEAzf38ZELA

https://www.911metallurgist.com/equipment/laboratory-jaw-crusher/

Function of key components and operating procedures for sample preparation equipment

- Rotary splitters
  - Designed for sample size reduction
  - Bulk sample added at top and then evenly distributed in sample compartments below
  - One or two portions are then taken for assay

https://geneq.com/materials-testing/en/product/sepor/rotary-sampler-splitter-701

Function of key components and operating procedures for sample preparation equipment

- Riffle splitters
  - Used to reduce a sample size into equal portions
  - Sample is reduced by half after each pass
  - Sample needs to be dry for splitting to be effective

https://www.researchgate.net/figure/Riffle-splitter-for-final-sampling_fig15_267858686

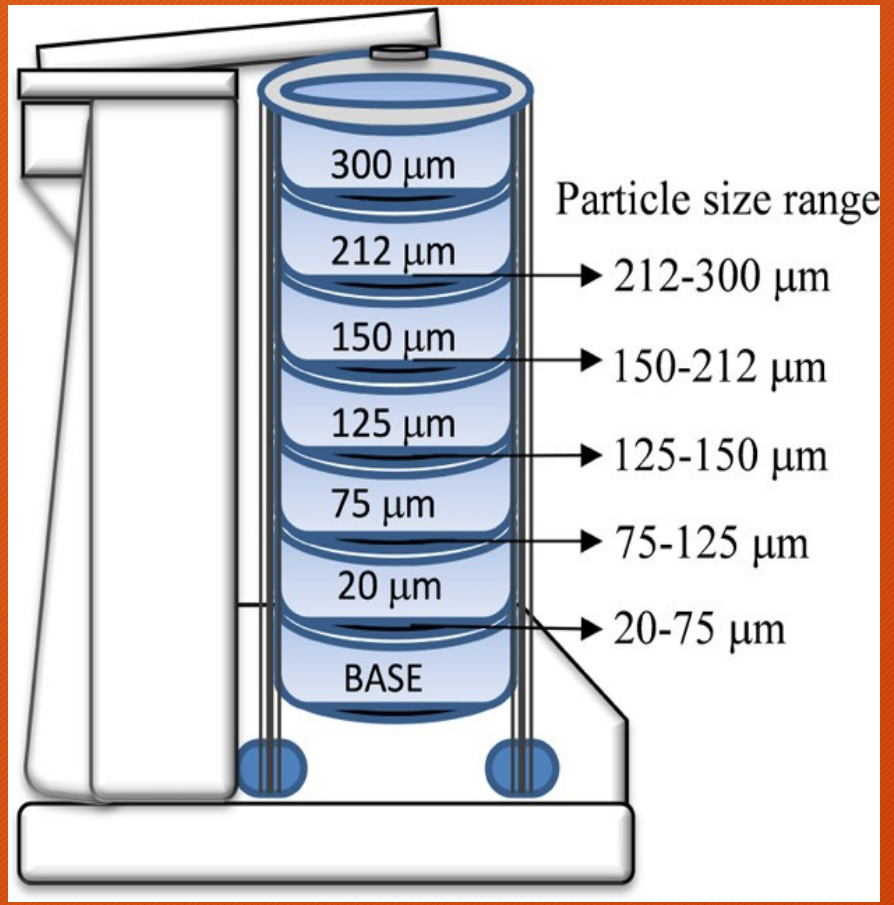
Function of key components and operating procedures for sample preparation equipment

- Ring mill pulveriser
  - Used as particle size reduction device
  - Samples pulverised typically after jaw crushing/ splitting
  - Ring mills come in different sizes depending on sample size
    - 1g-5kg
  - Possible to get to 75µm particle size depending on sample type and pulverising time
  - Video
    - https://www.youtube.com/watch?v=qsy17qGWlL4

https://www.savonaequipment.com/en/siebtechnik-ring-mill-pulverizer-p41903

Function of key components and operating procedures for sample preparation equipment

- Sieving
  - Process whereby a sample can be separated due to their particle size.
  - Depending on the client requirements, the sieving process can be done using a dry or wet sample, where the movement of particles is facilitated by the addition of water
  - The grouping of sieves together is termed 'a nest'



https://images.app.goo.gl/NJxdEQ33koCeQ9uJ6

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Sample Preparation – minimisation of contamination

- Minimisation of sample build up
  - Build up sample residues can occur on preparation equipment due to their physical nature or through compaction during the preparation process e.g. clays
  - Important to ensure all preparation equipment is free of sample prior to preparation of following sample
  - An air gun is used to remove excess dust and particulate matter on all equipment between samples to minimise contamination
  - A 'silica or quartz wash' is used to clean preparation equipment such as jaw crushers and pulverising bowls
  - In some cases, the pulverising times or the quantities being prepared have to be reduced to ensure the correct particle size can be obtained
  - Some laboratories (especially with robotic sample preparation) incorporate a water wash and drying step as part of the cleaning procedure

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Sample Preparation – minimisation of contamination

- Important to reduce the risk of sample contamination at all times in preparation of samples
- The reduction of sample build up on equipment is part of Good Laboratory Practice (GLP)
- Sample integrity must be maintained throughout all steps within the laboratory process, from sample receival through to sample disposal
- If there are any sample preparation difficulties encountered, a supervisor should be consulted in all circumstances

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Sample bagging & storage

- After drying, the samples are jaw crushed, split and pulverised (depending on the sample type) e.g. drill core requires jaw crushing, soils do not
- Once prepared, the fine powder is placed into sample bags and boxed ready for analysis within the laboratory
- The backup or unused portion of the samples are returned into their original calico bags and placed back into their plastic bags
- These are then returned to the boxed crate from which they were first delivered.
- Storage of the samples can be from 3-6 months, longer if required and costs involved due to restrictions on storage space at the laboratory

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Prepare Mineral Samples for Analysis

Sample digestion & fusions

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Sample Preparation methods - digestion

- Sample digestion/pre-treatment
  - partial digestion requiring separation (e.g. aqua regia)
    - Consists of 1 part HNO_3 and 3 parts HCl
    - Suitable for some elements of interest (e.g. Au and most base metals, Cu, Zn, Mn...)
    - $Au + 3HNO_3 + 4HCl \rightarrow HAuCl_4 + 3NO_2 + 3H_20$
    - Gold is digested from the sample and then solvent extracted into an organic solvent, such as di-isobutyl ketone (DIBK)
    - $CH_3CHCH_3CH_2COCH_2CH_3CHCH_3$ ($C_9H_{18}O$)

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Sample Preparation methods - digestion

- Complete digestion (e.g. multi-acid digest, including HF acid)
  - Required if a complete analysis is required for all elements potentially found in a sample
  - This requires a more aggressive approach to the digestion
  - This involves the use of a multi-acid digest, typically a mixture of the following reagents
    - Nitric acid, HNO_3
    - Bromine, Br_2
    - Perchloric acid, $HClO_4$
    - Hydrofluoric acid, HF
    - Hydrochloric acid, HCl

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Sample Preparation methods - digestion

- The method involves typically weighing 0.5g accurately into teflon beakers
- The acids are added in stages with variable digestion times for each on a moderate to high hot plate temperature, 150-200 degrees C.
- Total digest can take up to 8 hours to complete and is usually taken down to dryness
- The resultant residue is dissolved in di water and HCl and made up to a known volume, typically 100mL and then mixed thoroughly to ensure complete homogenisation of the sample.
- The samples are analysed using instrumental techniques, such as ICPOES, ICPMS or AAS depending upon the client request.

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Sample Preparation methods - fusions

- Fusions (e.g. Sodium peroxide, Na_2O_2)
  - Technique ideally suited to refractory type minerals (magnetite, ilmenite, chromite etc.), or minerals that don't respond well to conventional acid digestion methods
  - Samples are accurately weighed (typically 0.3-0.5g, dependent on the method and reagents used)
  - A pre-roast is done for approximately 30 minutes to remove unwanted volatile elements e.g. sulfur, arsenic. If these or any other volatile elements are required, a special acid digest would be required
  - Samples mixed with the peroxide and fused at around 680 degrees C
  - The peroxide method involves fusing in zirconium crucibles for 20 minutes before removing, cooling and placing the crucible and its contents into a beaker and adding water.
  - A violent reaction will occur with the metal analytes combining with the water to form insoluble oxides and hydroxides.
  - HCl is added in excess to dissolve the insoluble material which then passes into solution
  - The solution is then transferred to a volumetric flask and made to volume, mixed thoroughly and then an instrumental means (ICPOES/AAS) is made to determine the amount of metals found in the sample

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Sample Preparation methods - fusions

- NOTE: Sodium Peroxide, Na_2O_2 needs to be dry to be effective!
  - $2Na_2O_2 + 2H_2O \rightarrow 4NaOH + O_2$
  - Sodium peroxide is very reactive and absorbs moisture from the air to release oxygen gas
  - Good quality Na_2O_2 is a yellow granular powder. With time it turns white due to a mixture of sodium hydroxide and sodium carbonate being present
  - To prolong its shelf-life, Na2O2 should be stored in a desiccator

Sample Preparation methods - fusions

- Fusions (e.g. $LiF/Li_2B_4O_7$ discs or fused beads)
  - Finely ground samples (typically 75µM) are weighed into platinum crucibles
  - $LiF/Li_2B_4O_7$ is added (anywhere from 5:1 to 10:1, flux to sample ratio, depending on the method) and mixed well with the sample
  - A fusion is carried out between 900-1000 degrees C for approximately 30 minutes to 1 hour
  - Remove and let cool
  - The resultant glass bead is a homogeneous representation of the sample
  - The sample can either be:
    - transferred with the platinum crucible into a beaker and DI water and HCl added to dissolve the glass bead (NOTE: the Pt crucible does not dissolve in the acid)
    - The resultant solution is transferred to a volumetric flask and volumed to a known volume and read on an instrument such as an ICPOES/ICPMS or AAS to determine the concentration of the analyte/s in solution
  - OR:
    - The bead can be read directly using an alternative technology, such as an XRF spectrometer to determine the amount of analyte in the original sample

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Sample Preparation methods - fusions

- Disadvantages:
  - Costs to prepare sample
  - Time required
  - Costs of equipment e.g. Pt crucibles, fusion furnace, XRF/ICP's
  - Heat hazards
  - Poorer detection limits – small masses weighed to large volumes used
- Advantages:
  - High sample throughput
  - Accuracy
  - Good homogeneity of sample with little to no matrix effects

Sample Preparation methods – non-destructive techniques

- Non-destructive (pressed powder techniques)
  - Sample particle size ideally 75µM
  - Sample mixed with a grinding/binding agent (typically a cellulose/wax mixture of 20-30% of sample)
  - Mixture placed into a die and pressed at between 20-30 tonne to produce a homogeneous sample pellet
  - Sample is analysed using an XRF spectrometer

- Advantages:
  - Cheaper than the fused bead method
  - Potentially better detection limits due to no 'dilution' of sample
- Disadvantages:
  - Pressed powder is prone to matrix effects from a number of elements found in the sample

- Of the two techniques, the fused bead provides the greatest accuracy, as the sample produced is more homogeneous than the pressed powder

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Common analytes and chemical reactions

- Distribution of common analytes in a matrix
  - Knowledge of mineral content is useful in determining method selection for analysis of an ore
- Chemical reactions associated with common preparation methods used in job role
  - Use of aqua regia (HNO_3 and HCl)
  - Use of mixed acid (HNO_3, Br_2, $HClO_4$, HF, HCl)
  - Use of fluxes for fusions (sodium peroxide, Na_2O_2/lithium fluoride, LiF-lithium tetraborate, $Li_2B_4O_7$)
- Effects of reagents on the element of interest
  - E.g. combination of 1 part HNO_3 and 3 parts HCl to dissolve gold

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Common analytes and chemical reactions

- Reaction and recovery rates, solubility and equilibria
  - Awareness of types of reagents used when determining some analytes
    - E.g. use of HCl when determining silver or lead
      - $Ag^+ + Cl^- \rightarrow AgCl\ (s)$
  - Important to keep acid concentrations high to maintain analyte stability and equilibria in solution
- Tracking analytes of interest during changes of state
  - Be aware of the analyte being sought and possible changes that can occur during digestion
    - E.g. loss of gold as volatile gold chloride (Au_2Cl_6) during aqua regia digest
    - E.g. loss of volatile elements as oxides during roasting procedures (As, Se, Te, Hg, Sb, S etc.)
      - e.g. $2FeAsS + 5O_2 \rightarrow Fe_2O_3 + As_2O_3\ 2SO_2$
    - NOTE: smell of garlic during roasting of samples indicates presence of arsenic!

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Critical preparation steps that determine analytical accuracy & precision

- Monitoring drying (incipient & total)
  - Ensure samples are dry prior to subsampling
  - Ensure workplace SOP's are always followed and samples aren't digested to dryness, unless a SOP instructs otherwise (usually dependent on the element/s being determined)
- Mixing the sample to ensure homogeneity before sub-sampling
  - Prevent bias occurring during the subsampling step
- Suitability of reagents for purpose (e.g. dryness)
  - Samples must be dry prior to analysis to prevent errors in weighing
  - Chemical reagents must 'fit for purpose' and be moisture free to be effective for the method employed (e.g. sodium peroxide, Na_2O_2)
- Accurate operation of dispensers and balances
  - Minimise errors during volume addition to samples
  - Usually involves calibration of auto-dispensers

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Critical preparation steps that determine analytical accuracy & precision

- Critical/non-critical volumes and critical reagent quantities
  - Knowledge of what volumes are important during digests for method implementation
  - e.g. final volumes, serial dilutions
  - Some volumes are not critical, such as adding extra acid during a digest procedure.
    - In the formation of aqua regia (1 part HNO_3 + 3 parts HCl), extra HCl is added during the digest to prevent the samples drying out, which can lead to gold losses.
  - Any extra volume added would be driven off during the digest.
  - What is critical is the final volume the resultant solution is made up to, as this is required in the final concentration calculation in the sample

Critical preparation steps that determine analytical accuracy & precision

- Temperature control during digests
  - To avoid mechanical and chemical losses
  - Au can be lost as a volatile Au_2Cl_6 if samples taken to dryness
  - Sufficient digestion time is required to maximise analyte dissolution and for equilibrium to be reached.
    - This can be approximately 2 hours for an aqua regia digest (dependent on sample size) and up to 8 hours for a total digest using a multi acid digest
- Loss of solution prior to/after mixing
  - Incurs analytical losses affecting final result issued to client
- Type and acid strength in final solutions
  - Determines quality of digest and effectiveness of leaching of metal analytes into solution
    - e.g. the dissolving of Au in a sample is most effective using concentrated HNO_3 and HCl in a 1:3 ratio
    - solutions to be kept in an acid matrix to avoid loss of analytes at higher pH values (tendency to form insoluble oxides and hydroxides and pH's close to neutral and above)
- Mechanical loss of digest (sputtering, residues on glassware/plasticware and filtering)
  - Incurs analytical losses and potential for contamination between samples

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Prepare Mineral Samples for Analysis

Calculation steps & use of non-SI units

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Calculation steps

- Example 1 - Dilutions
  - $C_1V_1=C_2V_2$
  - E.g. calculate the volume required of 1000ppm Fe standard to make 250mL of 50ppm.
    - C_1= 1000ppm
    - V_1 = ?
    - C_2 = 50ppm
    - V_2 = 200mL

1000ppm x V_1 = 50ppm x 200mL

V_1 = (50ppm x 200mL)/1000ppm

V_1 = 10mL

Calculation steps

- Example 2 – Concentration
  - ppm to % calculation
  - Calculate the amount of Fe present in a sample if 0.5010g is digested in HCl and volumed to 500mL. The spectrometer gave a reading of 20.2ppm (20.2 μgmL^{-1})in solution. What is the %Fe in the original sample?
  - ppm Fe = (AAS result x Total volume)/Sample mass(g)
    - = (20.2μgmL^{-1} x 500mL)/0.5010g
    - = 20 160μgg^{-1}
    - = 20 160ppm
    - Now 10 000ppm = 1%
    - Therefore % Fe = 20160/10000 = 2.02%

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Use of non SI units and SI units

- S.I. definition
  - There are seven basic units in the SI system: the meter (m), the kilogram (kg), the second (s), the kelvin (K), the ampere (A), the mole (mol), and the candela (cd).

https://courses.lumenlearning.com/introchem/chapter/standard-units-si-units/#:~:text=There%20are%20seven%20basic%20units,and%20the%20candela%20(cd).

- Non S.I. units – ppm, ppb, g/L, %
  - Conversions:
    - ppm to ppb = x 1000 (1ppm = 1000ppb)
    - 1% = 10 000ppm
    - 1g/L = 1000ppm

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Use of non SI units and SI units

- Other non S.I. to S.I. conversions:

Non-SI term	Non-SI unit	Conversion	SI unit
minute	min	x 60	s
hour	h	x 3600	s
litre	l	x 10^{-3}	m^3
bar	bar	x 100	kPa
PSI	$lb.inch^{-2}$	x 6.895	kPa
atmosphere	atm	x 101.325	kPa
centigrade	°C	+273.15	K

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Prepare Mineral Samples for Analysis

Hazards

Hazards – sample preparation

- Hazards
  - Sample preparation – mineral dressing
    - Dust & fine particulates
    - Noise
      - Crushers
      - Pulverisers
      - Extraction systems
      - Air gun
    - Heavy containers
    - Manual handling & Lifting techniques
    - Slips, trips & falls
    - Moving equipment & parts
      - Rotary splitters
      - Jaw crushers
    - Electrical
      - Cords
      - 3-phase power
    - Sample types
      - Heavy metals (toxic) – nickel, arsenic
      - Radioactive - uranium
      - Asbestos - asbestosis risk
      - Silica - silicosis risk
      - Biological – bacteria
    - Chemicals & irritants

Hazards - Weighing

- Hazards
  - Sample preparation – weighing (top loader and/or analytical balance)
    - Sample types
      - Heavy metals (toxic) – nickel, arsenic
      - Radioactive - uranium
      - Asbestos - asbestosis risk
      - Silica - silicosis risk
      - Biological - bacteria

Hazards – Digestion techniques

- Hazards
  - Sample preparation – digestion
    - Use of acid mixtures
      - Partial sample digestion
        - Aqua regia digest - nitric (HNO_3), hydrochloric (HCl) (multi purpose general digest)
      - Complete sample digestion
        - 'Multi' acid digest - nitric (HNO_3), bromine (Br_2), perchloric ($HClO_4$), hydrofluoric (HF), hydrochloric (HCl)
      - Wet 'ashing' digest
        - For organic materials - nitric (HNO_3), perchloric ($HClO_4$) (for volatile element determinations)
        - Volatile elements – arsenic, antimony, mercury, selenium, tellurium, sulphur
      - NOTE: Dry ashing involves roasting of the samples in a furnace at 600 deg. C and digestion with nitric and/or hydrochloric acid. (This method is for non-volatile elements)

Hazards – reagents (refer to SDS!)

- Use of specialised reagents
  - Bromine (Br_2) (strong oxidizing reagent)
  - DIBK (di-iso butyl ketone)(for gold extraction in acid solution)
    - Flammable liquid
    - Irritant
  - Flocculating agents (to aid in the separation of solids from liquids)
  - Cyanide (for dissolution of gold)
    - PPE: safety glasses/lab coat/safety boots/self contained breathing apparatus/cyanide monitoring kit/fume cupboard
    - Antidote:
      - Cyanide Antidote Kit containing 2 Ampules Sodium Nitrite Injection (300 mg in 10mL of water) 2 Vials Sodium Thiosulfate Injection (12.5 g in 50mL of water) 12 Ampules Amyl Nitrite Inhalants (0.3 mL)
  - Hydrofluoric acid (for dissolution of silicates)
    - To be kept locked away when not in use
    - PPE & other requirements:
      - safety glasses/safety visor with neck protection/latex gloves/acid resistant gloves/safety boots/long sleeve shirt/lab coat/plastic sleeve protectors/long plastic apron/fume cupboard/serviced safety shower
    - Antidote:
      - calcium gluconate gel and injection kit in case of spill

Hazards - fusion

- Hazards
  - Sample preparation – fusion
    - Reagents
      - Sodium peroxide, Na_2O_2
      - Lithium fluoride, LiF
      - Lithium tetraborate, $Li_2B_4O_7$ / Lithium metaborate, $LiBO_2$
      - Sodium carbonate, Na_2CO_3
    - Heat
      - Use of high temperatures (700-1000 deg. C)
      - Emission of toxic volatile elements
        - Arsenic (smell of garlic), antimony, sulphur oxides, mercury, selenium, tellurium

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OSH – selected reagents

- Common reagents
  - Hydrochloric acid, (HCl) - corrosive
  - Nitric acid (HNO_3) – corrosive & oxidizing agent
  - Perchloric acid ($HClO_4$) - corrosive & strong oxidizing agent
  - Hydrofluoric acid (HF) - corrosive & toxic
  - Bromine (Br_2) - corrosive & strong oxidizing agent
  - DIBK (di-iso butyl ketone, $C_9H_{18}O$) – flammable & irritant
  - Sodium peroxide, (Na_2O_2) - strong oxidizing agent & corrosive
  - Cyanide (as NaCN or KCN) – highly toxic

- Remember the hierarchy of control
  - 1. Elimination
  - 2. Substitution
  - 3. Engineering controls
  - 4. Administrative controls
  - 5. PPE

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PPE & safety requirements – mineral dressing/sample preparation lab

- Dust/lab coat
- Safety boots
- Safety glasses
- Safety masks
- Ear protection
- Disposable gloves
- Extraction systems
- Manual handling aids such as trolleys

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PPE & safety requirements – digestion/fusion lab

- Lab coat
- Safety boots
- Safety glasses & visors – for acid digestion work
- Safety masks - if weighing toxic or hazardous materials
- Disposable gloves – heavy duty acid proof gloves when handling concentrated acids
- Extraction systems – fume cupboards
- Chemical antidotes for specific chemicals – e.g. cyanide & hydrofluoric acid
- Workplace SOPs to be at hand at all times
- Sufficient training to be provided to all staff prior to commencing work in the hazardous area –
  - NOTE: specialized training to be provided to staff using cyanides and hydrofluoric acid!

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Prepare Mineral Samples for Analysis

Environmental & workplace responsibilities

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Environmental issues

- Review information as outlined in SDS for waste disposal procedures and protocols
- Must follow guidelines as set out by local and state authorities for all types of hazardous wastes
- Follow all workplace SOPs and disposal protocols at all times for all forms of wastes, whether mineral or chemical in origin, solid or liquid.

- All laboratories must adhere to the Environmental Protection Act (waste to resources) Policy 2010 under the Environmental Protection Act 1993

Environmental issues – acidic and heavy metal wastes

- Common wastes
  - Acidic digest solutions
    - Low pH solutions
    - Holds metals in solution
  - Heavy metals
    - e.g. lead, mercury, arsenic, cadmium, nickel, copper, zinc, cobalt, chromium, manganese, vanadium….
    - Bioaccumulation risks
    - Mercury
      - Minamata disease
        - https://www.healthandenvironment.org/environmental-health/social-context/history/mercury-the-tragedy-of-minamata-disease
      - Karen Wetterhahn story
        - https://en.wikipedia.org/wiki/Karen_Wetterhahn
        - https://en.wikipedia.org/wiki/Dimethylmercury
    - Cadmium
      - Itai-itai disease
        - https://en.wikipedia.org/wiki/Itai-itai_disease

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Environmental issues – organic solvents

- Organic solvents (e.g. DIBK)
  - Generally flammable and irritants.
  - Many carbon based chemicals are classed as carcinogenic or dangerous to the major organs in the human body (e.g. TBE – tetrabromoethane used in heavy mineral separation)
  - Immiscibility with water
    - Do not dispose down sink!
  - Incompatible with waterways
    - Can impact ecosystems including flora & fauna

Workplace traceability

- Imperative to follow all workplace procedures for all samples submitted into a laboratory for analysis
  - Laboratories use reference equipment and standards to calibrate samples and/or equipment
  - This helps improve the confidence in the traceability of the data obtained
  - Both reference equipment and standards are usually traceable to a national standard e.g certified reference materials (CRM's)
- Legal and ethical requirements regarding traceability:
  - Chain of custody forms need to be followed at all times from sample receipt to disposal
  - All workplace data to be kept confidential at all times and no disclosure of any results to be issued to third parties without the express approval of the client
  - All records to be kept in a Laboratory Information Management System (LIMS) and backed up according to workplace procedures
  - Unused and backup samples to be retained by the laboratory until all analyses has been completed and stored from between 3 to 6 months, subject to client's instructions

Workplace responsibilities - employer

- To provide a safe working environment at all times for all employees
- To provide suitable PPE for all tasks required
- Ensure all equipment is operationally sound and fit for purpose
- Ensure all areas within the workplace are well maintained and
- Risks minimised or substituted wherever possible
- Where required, chemical safety including the safe use and storage of substances, SDS, PPE and equipment, first aid and location of safety showers and eye baths
- Employer to ensure adequate training and supervision is provided to all staff and for them to be deemed job ready prior to engaging in any duties performed
- A safety representative should be delegated and routine safety checks and inspections performed regularly. Formation of a safety committee is required and shift changover mmetings should be held daily to ensure adequate communication re workflows and safety issues
- Duty of care is the responsibility of all (employers and employees) for the safety of themselves and others in the workplace
- The OHS act of 1984 has now been superseded with the new act - Work Health & Safety (WHS) Act, 2020 coming into effect in 2022

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Workplace responsibilities - employee

- Ensure the safety of themselves and others at all times
- Follow all OHS/WHS procedures provided
- Follow all workplace SOP's & be familiar with (and know the location of) SDS information prior to commencing any procedure
- Co-operate with the employer in order to comply with OHS/WHS regulations at all times e.g. follow workplace policies and procedures
- Employees not to deliberately misuse equipment to ensure the health and safety of others around them
- Employees must report hazards and work-related injuries